

DIFERROCENYLS AND TERFERROCENYLS

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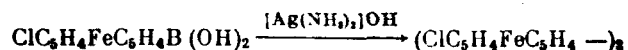
Methods for synthesizing a series of substituted diferrocenyls, 1,1'-diferrocenylferrocene (1,1'-terferrocenyl), and polyferrocenylenes are described in detail. The system of two linked cyclopentadienyl nuclei in the molecule of diferrocenyls absorbs in the infrared region near 1000 and 1113 cm^{-1} , which is close to the absorption frequencies of the unsubstituted cyclopentadienyl ring of ferrocene and its derivatives, showing that the Ullmann reaction proceeds without isomerization.

Recently, diferrocenyl has been synthesized by various methods. Thus, Nesmeyanova and Perevalova (Bibl.1) synthesized it by heating mercuric diferrocenyl with palladium; later Rausch used silver for this reaction (Bibl.2); as a by-product, diferrocenyl was isolated through interaction between a mixture of lithium- and 1,1'-dilithium ferrocenes and tri-N-hexylbromosilane (Bibl.3). We found that diferrocenyl can be synthesized along with ferrocene on interacting ferrocenylboric acid with an ammonia solution of silver oxide or a blue vitriol solution (Bibl.4). According to Perevalova and Nesmeyanova (Bibl.5) and the concurrent findings by Rausch (Bibl.2, 6), halogen ferrocenes enter into an Ullmann reaction with copper; the reaction produces high yields and gives diferrocenyl. The only derivatives of diferrocenyl known previously were benzoyldiferrocenyl and two isomeric diacetyldiferrocenyls (with unknown position of the substi-

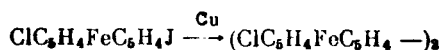
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tuent), obtained on acylating diferrocenyl (Bibl.2).

The present communication describes certain substituted diferrocenyls and 1,1'-diferrocenylferrocene (terferrocenyl). On reacting 1-(1'-chloroferrocenyl) boric acid with ammonia solution of silver oxide we succeeded in synthesizing bis[1'-(1'-chloroferrocenyl)]

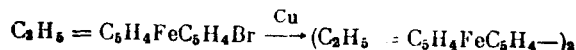


This dichlorodiferrocenyl is identical with the dichlorodiferrocenyl which we obtained from 1'-chloro-1-iodoferrocene by Ullmann reaction at 85°C:

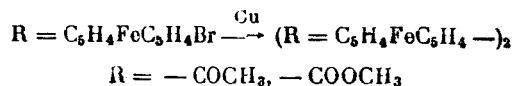


In this case, the chlorine linked to the ferrocene system is not affected; at this temperature, chloroferrocene does not react with copper while iodoferrocene gives diferrocenyl.

The Ullmann reaction also readily yields bis[1-(1'-ethylferrocenyl)] (120°C, 30 min):



Bromoferrocenes containing electronegative substituents ($-\text{COCH}_3$, $-\text{COOCH}_3$) react with copper much more slowly. However, on extending the reaction time (16 - 24 hrs, 125°C), the reaction proceeds with satisfactory yields



As the reaction temperature is increased, the reduction reaction of halogen ferrocenes becomes dominant. Thus, when 1'-bromo-1-acetylferrocene is heated with copper at 150°C for 30 min, acetylferrocene is obtained with an 80% yield. Bis [1-(1'-acetylferrocenyl)], M.P. 187 - 188°C also will form, with a yield of 46%, as the basic product of the acylation of diferrocenyl, along with small

amounts of acetyl- and triacetyldiferrocenyls, M.P. 137.5 - 138.5°C and M.P. 143 - 144°C, respectively. Since the melting point of the latter substance /668 is close to the melting point of one of the isomeric diacetyldiferrocenyls described by Rausch, while the analyses for carbon and hydrogen are similar for diacetyl- and triacetyldiferrocenyls, we made an analysis for oxygen and found the product to correspond to triacetyldiferrocenyl.

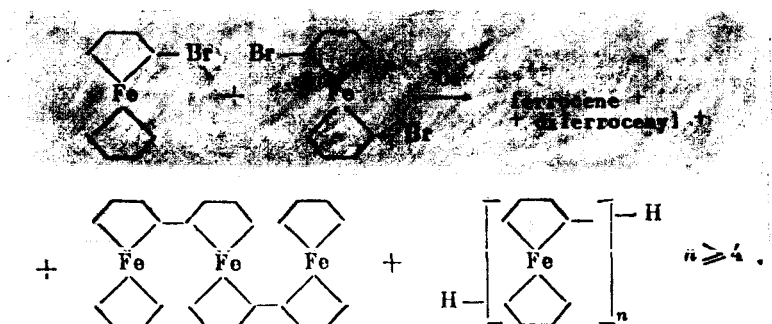
The infrared (IR) spectra of all disubstituted diferrocenyls have mean-intensity absorption bands of ~ 1000 and 1113 cm^{-1} , the first being weaker than the second. Ferrocene compounds with an unsubstituted cyclopentadienyl ring also have absorption bands of ~ 1000 and 1100 cm^{-1} (Bibl.7), but these usually are of greater intensity and the first is stronger than the second (cf. IR spectra). Despite these differences, the presence of the above-mentioned frequencies in the spectra of disubstituted diferrocenyls has raised some doubt as to the validity of the structures proposed above, since frequencies that are highly similar are typical of the derivatives of a ferrocene with an unsubstituted ring. It might be assumed, for example, that all the reactions that yield diferrocenyls involve isomerization, resulting in the formation of isomeric disubstituted diferrocenyls with nonsubstituted cyclopentadienyl rings, although so far no isomerization has been observed in an Ullmann reaction. On the other hand, thin-section chromatography of the products of the chlorination of bis[1-(1'-acetylferrocenyl)] and bis[1-(1'-carbomethoxyferrocenyl)] on silica gel in heptane revealed no pentachlorocyclopentane although one of us [Perevalova and her coworkers (Bibl.8)] established that ferrocene compounds containing nonsubstituted cyclopentadienyl nuclei disintegrate on halogenation and form pentahalocyclopentanes. The validity of the structure proposed by us for bis[1-(1'-ethylferrocenyl)] has been confirmed by its synthesization from bis[1-(1'-

acetylferrocenyl)] through Clemmensen reduction.

A radiographic analysis of dichlorodiferrocenyl, performed by Struchkov, Kaluskiy, and Avoyan, showed that the compound crystallizes in a monoclinic crystal system, space group $P2_1/c$ with two molecules in a cell. In this space group, the factor 2 corresponds to a particular position of the centers of symmetry, i.e., a molecule of this substance has a center of symmetry, which confirms the structure we propose. Thus, the system of two connected cyclopentadienyl nuclei in the molecule of diferrocenyls absorbs in the infrared region near 1000 and 1113 cm^{-1} , which is close to the absorption frequencies of the unsubstituted cyclopentadienyl ring of ferrocene and its derivatives; thus, it may be concluded that the Ullmann reaction in the ferrocene series proceeds in the standard manner, without isomerization.

Earlier it was noted (Bibl.9) that the polyferrocenylenes synthesized from ferrocene by polyrecombination reaction display ferromagnetic properties, whereas diferrocenyl and ferrocene are diamagnetic. Therefore, it was of interest to synthesize polyferrocenylenes of a definite structure from halogen ferrocenes by the Ullmann reaction and to investigate their properties.

We found that, due to the interaction between a mixture of bromoferrocene and 1,1'-dibromoferrocene with copper at 105 - 120°C, following chromatography on aluminum oxide, we were able to isolate diferrocenyl, 1,1'-diferrocenyl-ferrocene(1,1'-terferrocenyl), and the higher homologs from the reaction products



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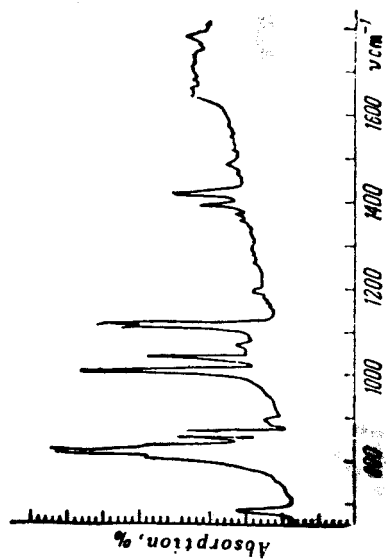


Fig.1 Diferrocenyl

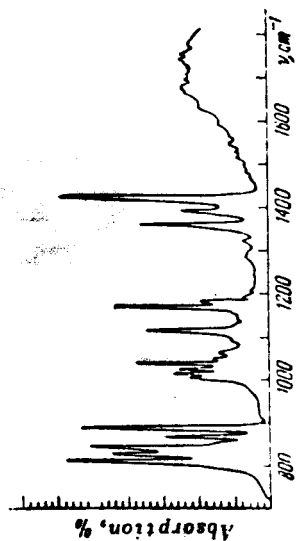


Fig.2 Bis[1-(1'-chloro-ferrocenyl)]

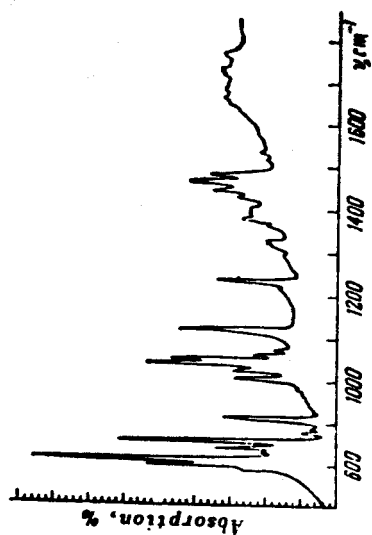


Fig.3 Bis[1-(1'-ethyl-ferrocenyl)]

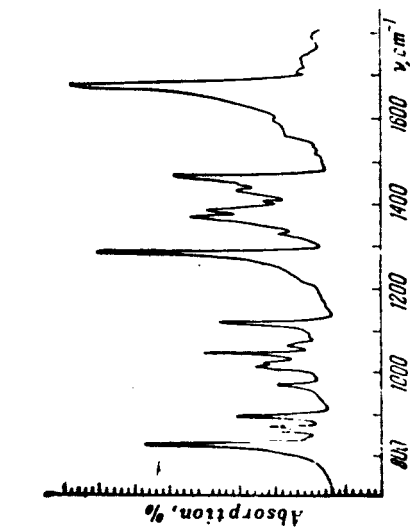


Fig.4 Bis[1-(1'-acetyl-ferrocenyl)]

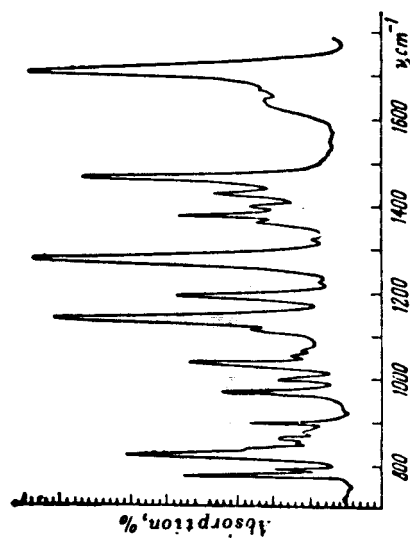


Fig.5 Bis[1-(1'-carbo-methoxyferrocenyl)]

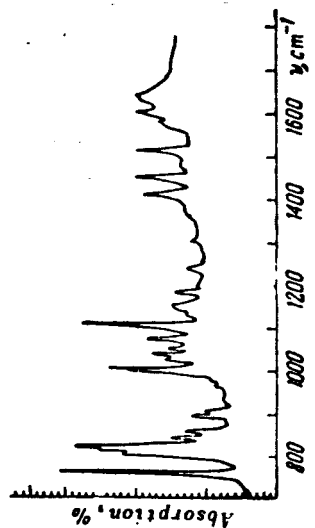
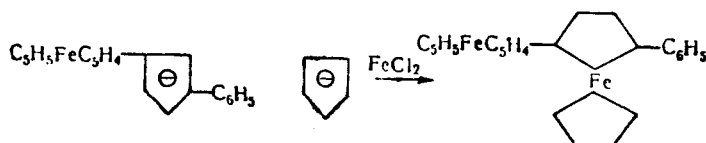


Fig.6 1-Ferrocenyl-3-phenylferrocene

Al_2O_3 and CaCO_3 .

On carrying out a reaction of the same kind in the presence of cyclopentadiene, 1-ferrocenyl-3-phenylferrocene was synthesized



along with ferrocene and 1,1'-diferrocenyl-3,3'-diphenylferrocene. Infrared spectra of diferrocenyl, substituted diferrocenyls, and 1,1'-terferrocenyl are presented in Figs.1 - 7.

Experimental Part

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Bis[1-(1'-chloroferrocenyl)]. a) From 1-(1'-chloroferrocenyl)boric acid:

An ammonia solution of 0.45 gm AgNO_3 is added to a hot solution of 0.7 gm 1-(1'-chloroferrocenyl)boric acid (Bib1.12) while stirring. After slow cooling, the reaction mixture is extracted with ether, the ether extract is washed with

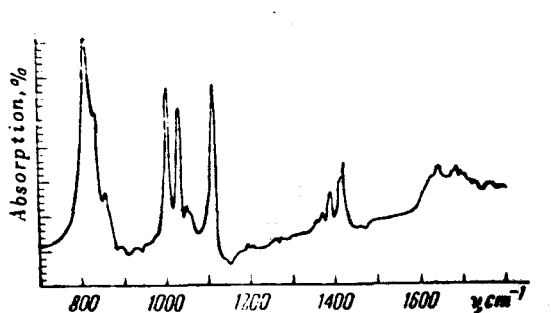


Fig.7 1,1'-Diferrocenylferrocene
(1,1'-terferrocenyl)

10% KOH and then with water, filtered to separate it from the silver, and the ether is driven off. The residue is subjected to Al_2O_3 chromatography; elution with petroleum ether yields 0.11 gm (22% of theoretical) chloroferrocene, M.P. 56 - 57°C; there is no decrease in melting point with respect to the a priori

known chloroferrocene. The benzene fraction yielded 0.13 gm (26% of theoretical) bis[1-(1'-chloroferrocenyl)], M.P. 136.5 - 137°C (from absolute alcohol). Found: C 54.76; 54.72; H 3.73; 3.73; Cl 16.28; 16.28; Fe 25.47; 25.70%. $C_{20}H_{16}Fe_2Cl_2$. Calculated: C 54.72; H 3.67; Cl 16.16; Fe 25.45%.

b) From 1'-chloro-1-iodoferrocene: A mixture of 0.34 gm 1'-chloro-1-iodoferrocene (Bibl.12) is heated with 1 gm copper (in a nitrogen stream) at 80-85°C for 30 min and, after cooling, washed repeatedly with ether. The ether is driven off, and the residue is subjected to aluminum oxide chromatography. The benzene fraction yields 0.19 gm (88% of theoretical) bis[1-(1'-chloroferrocenyl)], M.P. 134 - 135°C (from absolute alcohol), identical with that obtained from 1-(1'-chloroferrocenyl)boric acid (melting point of mixed sample, IR spectrum).

Bis[1-(1'-ethylferrocenyl)]. A mixture of 0.4 gm 1'-bromo-1-ethylferrocene (Bibl.13) with 1 gm copper is heated at 125°C for 30 min and, after cooling, repeatedly washed with ether. The ether is driven off, and the residue is subjected to aluminum oxide chromatography. The benzene fraction yields 0.29 gm (quantitatively) of bis[1-(1'-ethylferrocenyl)], M.P. 90 - 91°C; after recrystallization from alcohol, M.P. 96.5 - 97°C. Found: C 67.51; 67.57; H 6.29; 6.26; Fe 25.97; 26.04%. $C_{24}H_{26}Fe_2$. Calculated: C 67.64; H 6.15; Fe 26.21%.

Acylation of diferrocenyl. The reaction is accomplished in a dry nitrogen stream. A mixture of 0.53 gm aluminum chloride and 0.32 cc acetyl chloride in 15 cc absolute methylene chloride is poured into a three-necked flask equipped with a stirrer, a tube for the introduction of nitrogen and a tube for calcium chloride, after which 0.60 gm diferrocenyl is added. After stirring for 2 hrs at room temperature, the mixture is poured into ice water acidified with HCl and then extracted with chloroform. The chloroform extracts are washed with water

and dried with MgSO_4 , the solvent is driven off, and the residue is subjected to aluminum oxide chromatography. Benzene is used for eluting an insignificant amount of unreacted diferrocenyl, after which acetyldiferrocenyl 0.09 gm (13.5% of theoretical), M.P. $137.5 - 138.5^\circ\text{C}$ (in a nitrogen stream) crystallizes from heptane. Found: C 64.36; 64.22; H 5.03; 4.95; Fe 26.86; 26.85%. $\text{C}_{22}\text{H}_{20}\text{Fe}_2\text{O}$. Calculated: C 64.12; H 4.89; Fe 27.11%.

A benzene-chloroform mixture (7:3 by volume) eluates bis[1-(1'-acetylferrocenyl)], 0.34 gm (46% of theoretical), M.P. $187 - 188^\circ\text{C}$ (under nitrogen), crystallizing from a heptane-toluene mixture. Found: C 63.69; 63.68; H 4.93; 4.92; Fe 24.37; 24.37%. $\text{C}_{24}\text{H}_{22}\text{Fe}_2\text{O}_2$. Calculated: C 63.47; H 4.88; Fe 24.60%. 1672

Chloroform eluates triacetyldiferrocenyl, 0.05 gm (6.2% of theoretical), M.P. $143 - 144^\circ\text{C}$ (from a heptane-toluene mixture). Found: C 63.39; 63.32; H 5.04; 4.96; Fe 22.46; 22.32; O 10.01; 9.94%. $\text{C}_{26}\text{H}_{24}\text{Fe}_2\text{O}_3$. Calculated: C 62.93; H 4.88; Fe 22.50; O 9.68%.

Bis[1-(1'-acetylferrocenyl)]. A mixture of 0.5 gm 1'-bromo-1-acetylferrocene (Bibl.13) and 1 gm copper is heated in a sealed ampoule at 125°C for 24 hrs and, after cooling, washed with hot benzene. The benzene is driven off and the residue subjected to aluminum oxide chromatography. Elution with benzene yields acetylferrocene, 0.08 gm (21.5% of theoretical), M.P. $84 - 86^\circ\text{C}$. A mixed sample containing known acetylferrocene will melt at the same temperature. Chloroform eluates upward of 0.20 gm bis[1-(1'-acetylferrocenyl)] (54% of theoretical), M.P. $188 - 188.5^\circ\text{C}$ (under nitrogen), crystallizing from a heptane-benzene mixture. A mixed sample containing bis[1-(1'-acetylferrocenyl)], obtained by acylation of diferrocenyl, does not depress the melting point.

Reduction of bis[1-(1'-acetylferrocenyl)] by the Clemmensen method. Zinc dust (1 gm) is amalgamated with 0.1 gm of corrosive sublimate in 4 cc diluted

HCl (1:20). After 10 min, the liquid is decanted, and a solution of 0.10 gm of bis[1-(1'-acetylferrocenyl)] in 2.5 cc acetic acid and 2.5 cc conc. HCl are added. The mixture is carefully boiled until disappearance of the dark-cherry ketone color (5 min), cooled, filtered to remove the zinc, under elution of the zinc by ether. The ether solution is washed with water, 10% KOH, and water again, then dried with MgSO_4 ; the ether is driven off, and the residue subjected to aluminum oxide chromatography. Elution with heptane yields 0.04 gm (43% of theoretical) bis[1-(1'-ethylferrocenyl)], M.P. 93 - 94°C (from alcohol); in a sample known to contain this substance, there was no depression of the melting point.

Bis[1-(1'-carbomethoxyferrocenyl)]. A mixture of 0.97 gm methyl ether of 1'-bromoferrocene-1-carboxylic acid (Bibl.14) and 2 gm copper is heated in a sealed ampoule at 125°C for 16 hrs and, after cooling, washed with hot benzene; the benzene is driven off, and the residue subjected to aluminum chloride chromatography. Elution with benzene yields 0.10 gm (14% of theoretical) methyl ether of ferrocenecarboxylic acid; in a sample known to contain this substance, there was no depression of the melting point. A chloroform-ether mixture eluates bis[1-(1'-carbomethoxyferrocenyl)], 0.52 gm (71.5% of theoretical), M.P. 184 - 184.5°C (under nitrogen), crystallizing from a heptane-benzene mixture. Found: C 59.58; 59.77; H 4.66; 4.67; Fe 22.29; 22.23%. $\text{C}_{24}\text{H}_{22}\text{Fe}_2\text{O}_4$. Calculated: C 59.29; H 4.56; Fe 22.98%.

Unsubstituted cyclopentadienyl ring test for derivatives of ferrocene and diferrocenyl. The investigated substance (40 mg) is dissolved in 4 cc dry chloroform, and a stream of dry chlorine is passed through the solution for 45 min. Then the chloroform is driven off, the residue dissolved in ether, the ether solution washed with water (from FeCl_3) and dried with MgSO_4 . The ether

is driven off, the residue is dissolved in a drop of benzene and subjected to chromatography on a plate coated with silica gel; heptane is used as the eluant and pentachlorocyclopentane as the standard spot. Developing agent: iodine.

The known pentachlorocyclopentane standard shows $R_f = 0.36 - 0.40$. It is readily detected in products of the chlorination of acetylferrocene and methyl ether of ferrocenecarboxylic acid, but is absent in products of the chlorination of bis[1-(1'-acetylferrocenyl)] and bis[1-(1'-carbomethoxyferrocenyl)].

1,1-Diferrocenylferrocene (1,1'-terferrocenyl) and higher 1,1'-polyferrocenylenes. a) A mixture of 1 gm 1,1'-dibromoferrocene, 0.78 gm bromoferrocene, and 3.56 gm copper is heated for 15 min at 105°C, after which the temperature is gradually raised to 120°C over a period of 45 min and the heating is continued at this temperature for 30 min. After cooling, the reaction mixture is washed with benzene, the benzene is driven off, and the residue is subjected to chromatography in an aluminum chloride column (layer length 57 cm, d = 3 cm). Elution with heptane initially yields a mixture of ferrocene and halogenferrocenes - 0.23 gm, and later 0.40 gm (57% of theoretical, calc. in bromoferrocene) diferrocenyl, while elution with a mixture of 5 parts of benzene to 95 parts of heptane yields 0.17 gm (14% of theoretical, calc. in 1,1'-dibromoferrocene) 1,1'-diferrocenylferrocene(1,1'-terferrocenyl), and subsequently benzene eluates the higher polyferrocenylenes (0.13 gm).

The 1,1'-terferrocenyl is a yellow crystalline substance readily soluble in benzene, toluene, and chloroform and less soluble in carbon tetrachloride, heptane, alcohol. It crystallizes from heptane or alcohol, and may be driven off in vacuo at 240°C (7 mm) under nitrogen; M.P. 212.5 - 214.5°C (under nitrogen); the melting point is depressed with diferrocenyl.

The molecular weight of 1,1'-terferrocenyl was determined by mass spec-

trometry as 554 and by X-ray analysis as 529.5, compared with a calculated value of 554. Found: C 65.18; 65.12; H 4.69; 4.71; Fe 29.98; 29.80%. $C_{30}H_{28}Fe_3$. Calculated: C 65.03; H 4.73; Fe 30.24%.

Polyferrocenylenes are purified by reprecipitation from benzene with petroleum ether; decomposition temperature 260 - 265°C (under nitrogen). Found: C 65.96; 65.95; H 4.72; 4.84; Fe 29.20; 29.10%. $(C_{10}H_8Fe)_n$. Calculated: C 65.26; H 4.38; Fe 30.36%.

b) Analogously, a mixture of 2.30 gm bromoferrocene, 1.5 gm of 1,1'-dibromoferrocene and 7.6 gm copper yields 1.06 gm (66.5% of theoretical, calculated in bromoferrocene) diferrocenyl, 0.40 gm (16.5% of theoretical calculated in 1,1'-dibromoferrocene) 1,1'-terferrocenyl, and 0.23 gm higher polyferrocenylenes.

c) Under similar conditions, 0.80 gm 1,1'-dibromoferrocene and 1.6 gm copper yields 0.11 gm of a mixture of ferrocene and halogenferrocene, 0.09 gm (14% of theoretical) diferrocenyl, 0.03 gm (7% of theoretical) 1,1'-terferrocenyl and 0.05% gm higher polyferrocenylenes.

3-Ferrocenyl-1-phenylcyclopentadiene. The reaction is carried out in a stream of dry nitrogen. A total of 10.8 gm ethyl ether of β -benzoylpropionic acid is added to dry sodium ethylate obtained from 3 gm sodium, in 160 cc absolute benzene. The mixture is stirred for 15 min, after which 12 gm acetylferrocene are added and the mixture is left standing for 24 hrs, being heated on the water bath at 40°C for the last 5 hrs of that time. The flask is then cooled, the mixture poured into ice water (~400 cc), the water layer is decanted, washed with ether, and heated on the water bath at 70°C for 35 min. The precipitated red crystalline residue is filtered, washed with water, and dried in air. Yield: 3 gm 3-ferrocenyl-1-phenylcyclopentadiene; by ether extraction of the aqueous solution an additional 0.6 gm can be recovered. Total yield:

3.6 gm (21% of theoretical). The product is readily soluble in organic solvents, crystallizes from aqueous acetone and has a M.P. of 102 - 104°C. Found: C 77.17; 77.20; H 5.47; 5.66; Fe 17.07; 17.13%. $C_{21}H_{18}Fe$. Calculated: C 77.32; H 5.56; Fe 17.12%.

1,1'-Diferrocenyl-3,3'-diphenylferrocene. A total of 0.75 gm 3-ferrocenyl-1-phenylcyclopentadiene is added to sodium amide (0.13 gm sodium in 30 cc liquid ammonia), and the mixture is stirred for 2 hrs. After this and on cooling with solid carbon dioxide and acetone, 0.75 gm anh. ferric chloride and 20 cc xylene are added and the mixture is let stand overnight under nitrogen. The residue is filtered, washed with hot benzene directly on the filter (until colorless), the solvent is driven off, and the residue is subjected to aluminum oxide chromatography. Elution with a mixture of heptane and benzene (7:3 by volume) yields 0.18 gm (22% of theoretical) 1,1'-diferrocenyl-3,3'-diphenylferrocene, M.P. 232 - 233°C (under nitrogen), crystallizing from a heptane-benzene mixture. During chromatography, the aluminum oxide darkens intensely, possibly owing to 674 partial decomposition of the substance. Found: C 71.47; 71.47; H 5.00; 5.07; Fe 23.57; 23.53%. $C_{42}H_{34}Fe_3$. Calculated: C 71.42; H 4.85; Fe 23.72%. Molecular weight, as experimentally determined by isothermal distillation, was 738 against the theoretically calculated 706.

1-Ferrocenyl-3-phenylferrocene. A total of 0.50 gm 3-ferrocenyl-1-phenylcyclopentadiene is added to sodium amide (0.64 gm sodium in 40 cc liquid ammonia) and the mixture is stirred for 30 min; after this, 0.63 cc freshly distilled cyclopentadiene is added and the mixture is stirred for an additional hour. Subsequently, on cooling with solid carbon dioxide and acetone, 2.3 gm anh. ferric chloride and 25 cc xylene are added and the mixture is let stand overnight under nitrogen. The residue is filtered, washed with hot benzene (until colorless),

the solvent is driven off, and the residue subjected to aluminum oxide chromatography. Elution with heptane yields 0.54 gm (77% of theoretical, calc. in cyclopentadiene) ferrocene, while elution with a heptane-benzene mixture (7:3 by volume) yields 1-ferrocenyl-3-phenylferrocene (0.1 gm, 15% of theoretical) and, subsequently, 1,1'-diferrocenyl-3,3'-diphenylferrocene (0.09 gm, 16.5% of theoretical). 1-Ferrocenyl-3-phenylferrocene is a yellow crystalline substance of M.P. 172.5 - 173 °C (under nitrogen), which crystallizes from heptane. Found: C 70.03; 69.85; H 4.99; 4.99; Fe 25.02; 25.06%. $C_{28}H_{22}Fe_2$. Calculated: C 69.99; H 4.97; Fe 25.04%.

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Conclusions

1. A series of substituted diferrocenyls, 1,1'-diferrocenylferrocene(1,1'-terferrocenyl), and polyferrocenylenes were synthesized.

2. The frequencies of 1000 and 1113 cm^{-1} recorded for the spectra of the disubstituted diferrocenyls lacking a free cyclopentadiene ring are inherent to a system of interconnected cyclopentadienyl nuclei.

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